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The objectives of the project were to synthesize and characterize a polymer which could be switched to its conductive state by intramolecular electron transfer initiated by absorption of light. By covalently bonding the electron donating moiety to the polymer, the rate of switching should be fast and uniform giving it potential applications in optical processing devices. The initial polymer system selected was a polythiophene substituted at the 3 position with a diphenyliodonium salt. Photodissociation of diphenyliodonium chloride in physical contact with the polymer formed the highly oxidizing phenyliodonium radical cation which in turn oxidized the polythiophene backbone. Several approaches tried to synthesize the covalently bound substituted polythiophene were unsuccessful. Polymerization of 3-ferrocenylpyrrole, which also has potential photodoping properties, was also unsuccessful.

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"Synthesis and Oxidation Potential of 3-Ferrocenylpyrrole," T. L. Rose and A. B. Kon, Inorg. Chem., 32, xxxx (1993).

ABSTRACT OF OBJECTIVES AND ACCOMPLISHMENTS:

The objectives of the project were to synthesize and characterize a polymer which could be switched to its conductive state by irradiation with light. The rate of switching should be enhanced by covalently bonding a photoactivated electron transferring moiety directly to the polymer. The specific goals of the project were (1) to synthesize a polythiophene system with a covalently attached iodonium salt, (2) to illuminate the polymer to induce a persistent photochromic and photoconductive effect, and (3) to measure the optical constants of the film resulting from the photodoping. Several different approaches were unsuccessful in synthesizing a conductive polymer with a covalently substituted photoactive group presumably because of the bulky size of the chromophore. Demonstration of the concept of fast optical switching of polymers of this type, therefore, will require overcoming the synthetic difficulties.

Attempts to synthesize the 3-substituted iodonium thiophene monomer were unsuccessful because of the high reactivity of the 2-position. As an alternative route a copolymer of 3-methylthiophene and 2-(3-thienyl)ethyl phenolate was formed by electrochemical polymerization. This copolymer could be photodoped to its oxidized state by immersing it in an iodonium chloride solution followed by irradiation with deep UV light. However, treatment of the copolymer film with iodosobenzene diacetate rather than the iodonium chloride did not convert the phenolate to the desired covalently bound iodonium compound, and no photodoping was observed when the treated polymer was illuminated.

The UV-vis spectrum was measured for 3-ferrocenylpyrrole, another system which might undergo photoswitching when incorporated into a polymer. The spectrum indicated significant electronic interaction between the pyrrole and ferrocene with a large increase in the intensity of the bands attributed to charge transfer transitions. Thus far, however, 3-ferrocenylpyrrole has resisted polymerization by electrochemical and chemical means.

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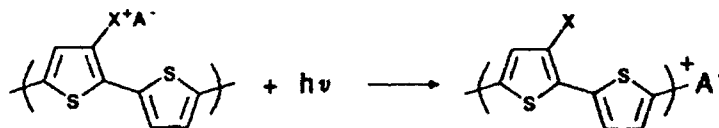
1.0 INTRODUCTION

In response to the growing need for increased information storage capacity and computing speed, new technologies are being developed based on processing by optical rather than electronic means. Optical processing is many times faster than electronic and is not susceptible to electromagnetic interference either by outside sources or internal cross-talk. We investigated the synthesis and characterization of a new material in the class of organic conductive polymers with the potential for photoswitching from an insulating to a conductive state.

Conducting polymers are of current interest because they combine mechanical and chemical properties of polymers with the electrical properties of metals [1]. One aspect of conductive polymers which distinguishes them from metals is their ability to switch reversibly between an insulating and conductive state. The switch between the reduced, neutral state and the oxidized, conductive state of the polymer is generally done electrochemically as indicated below for polythiophene.



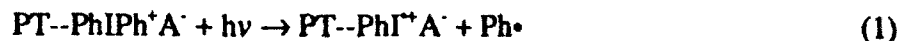
To retain an overall neutral system, the counter ion A^- diffuses in and out of the polymer structure during the switching. In the present application, we will switch the polymer using light, and thus the system falls into the general category of photochromic materials. The light is coupled into the polymer by forming the polymer from monomers with a covalently attached photosensitive salt. Light absorbed by the chromophore induces an electron transfer to oxidize the polymer chain.



In previous studies of photodoping, the material absorbing the light was either in a film above the polymer [2], diffused from solution into the polymer [3,4], or mixed with the polymer [5]. The approach investigated in this work represents the first attempt to attach the chromophore covalently to the polymer backbone. Attachment should optimize the switching rate and efficiency of the photodoping process due to the spatial proximity of the donor and acceptor since rate constants for electron transfer are a function of their separation.

For this program we initially investigated a polythiophene with an attached diphenyliodonium moiety. Laser flash photolysis studies of diphenyliodonium showed that the initial products are formed by homolytic cleavage of a Ph-I bond to give the phenyliodonium radical cation ($\text{PhI}^{\bullet+}$) and a phenyl radical [6]. $\text{PhI}^{\bullet+}$ is a strong oxidizing agent that will oxidize the thiophene system. The fate of the phenyl radical is unclear. Several reactions are possible: cage recombination with $\text{PhI}^{\bullet+}$,

which would reduce the efficiency of the doping reaction; reaction with a second phenyl radical to form biphenyl; reaction with the thiophene or phenyl group of the diphenyliodonium compound to release a proton. The general overall reaction scheme for our iodonium salt substituted polythiophene (PT--PhI⁺Ph⁻A⁻) is as follows:



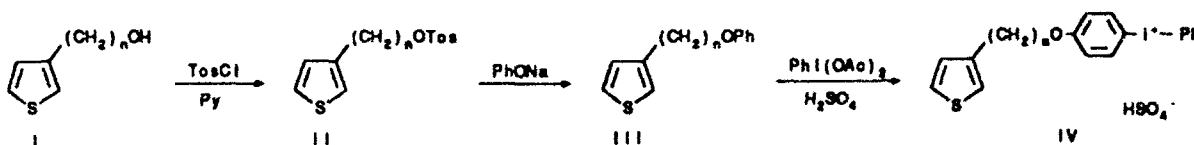
The overall goal of the research program is to demonstrate a new polymeric material that can be used for switching applications in optical processing. The initial study was designed to answer the question of whether a photoinduced reaction can lead to rapid doping by intramolecular charge transfer and had the following specific technical objectives.

- Synthesize a polythiophene with a covalently attached iodonium salt.
- Demonstrate that illumination of the polymer induces a persistent photochromic and photoconductive effect.
- Measure the change in the optical constants of the film resulting from the photolytic doping.

2.0 RESULTS

Synthesis of Thiophene Monomers

The synthesis of the thiophene iodonium monomer, **IV** ($n=1$), was attempted according to Scheme I. Formation of the tosylate from the starting alcohol, 3-thiophenemethanol, **I** ($n=1$), however, by the reaction of tosylchloride in a pyridine solution was unsuccessful. Others have also reported difficulty in forming benzyl tosylates which are unstable and cannot be successfully stored for prolonged periods [7].



Scheme I

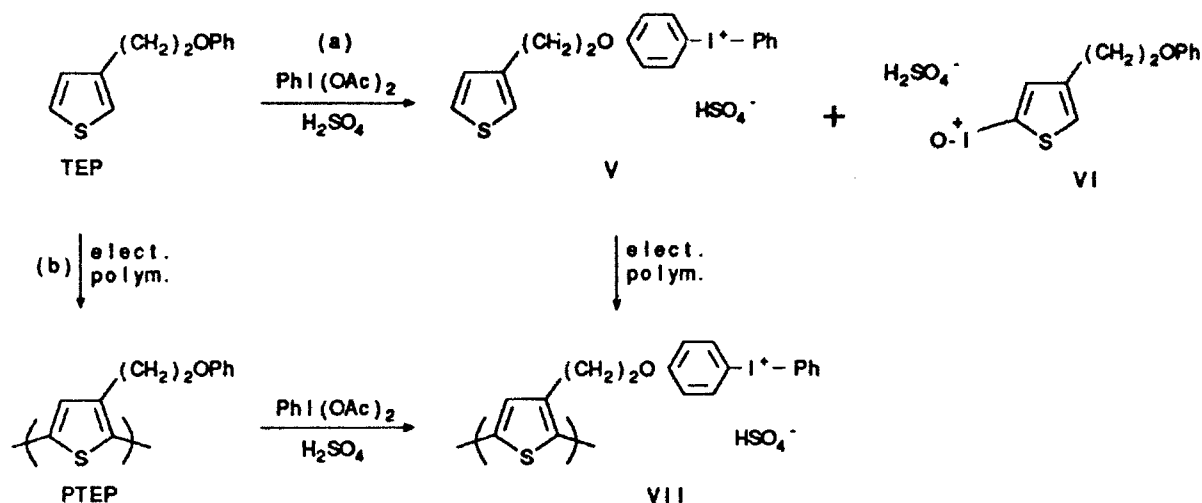
The synthesis was repeated using 2-(3-thienyl)ethanol, **I** ($n=2$), as the starting material. The intermediate tosylate, **II** ($n=2$), identified by the IR spectrum, was isolated with 70% yield as a white crystalline solid with a melting point of 50-51°C. **II** ($n=2$) was reacted in an aqueous solution of phenol with sodium hydroxide to give a colorless liquid 2-(3-thienyl)ethyl phenolate (TEP), **III** ($n=2$), in 60% yield. IR (NaCl plates) 3104, 3061, 3040, 2926, 2872, 1599, 1499, 1472, 1385, 1302, 1246, 1172, 1080, 1038, 839, 777, 756, 693, 639 cm^{-1} . NMR (CDCl_3) δ 3.16 (t, 2H), 4.17 (t, 2H), 6.92 (m, 3H), 7.03 (m, 1H), 7.08 (m, 1H), 7.26 (m, 3H).

TEP was treated with iodosobenzene diacetate according to the procedure for making unsymmetrical iodonium compounds described by Crivello and Lam as shown in Scheme II, path (a) [8]. Instead of the desired white crystalline compound, 4-(3-thienyl)ethoxydiphenyliodonium bisulfate, **V**, a cream colored micell-like dispersion was formed. We did not identify the product(s), but we suspect that they are short-chain oligomers of poly-TEP and/or the iodoso compound **VI** resulting from reaction at the more reactive 2-position of the thiophene.

Electropolymerization of 2-(3-thienyl)ethylphenolate (TEP)

As an alternative to synthesizing the iodonium substituted thiophene monomer **V** followed by electropolymerization, we investigated the route of first electropolymerizing the TEP and then reacting the polymer with the iodosobenzene diacetate reagent (Scheme II, route (b)). Using this route, the 2 position of the thiophene ring would be blocked before addition of the iodosobenzene diacetate. TEP was electropolymerized in a three electrode electrochemical cell with dry acetonitrile (ACN) as solvent and tetrabutylammonium tetrafluoroborate (TBATFB) as electrolyte. The working electrode was a polished Pt disk (area = 0.03 cm^2) sealed in glass. The reference electrode was Ag/Ag(NO_3) (0.1M) and the counter electrode a large area Pt foil. $E_{p/2}$ for a 0.02M

ferrocene/ACN solution was 0.050V vs Ag/Ag(NO₃) at a scan rate of 100 mV/s. All potentials are given vs the Ag/Ag(NO₃) reference electrode which had a potential of about 0.35V vs SCE. The electrochemical experiments were conducted in an Ar filled dry box.



Scheme II

The $E_{p/2}$ for oxidation of 0.0025M TEP in 0.4M TBATFB/ACN was 1.37V vs at 100 mV/s. This potential is somewhat lower than that reported for oxidation of other thiophenes with an ether substituent at the 3 position [9]. Continuous cycling between 0V and 1.5V lead to growth of a film of poly(2-(3-thienyl)ethyl phenolate) (PTEP). With successive sweeps, however, the oxidation potential of PTEP moved to more positive values. The peak current increased for the first 4 or 5 sweeps and then decreased. The peak potential for the oxidation of the polymer was 0.82V for the fourth cycle. There was a broad cathodic wave at $\approx 0.8V$ with a peak current only 1/10 the peak current of the anodic wave. A film grown by cycling the electrode for three cycles was transferred to a solution of 0.1M LiClO₄ in ACN. The first sweep at 100 mV/s had an anodic peak at 0.71V, and the film changed color from yellow in the reduced form to a green-black at 1.0V. Only about 1/5 of the charge was recovered in the return cathodic sweep which had a broad peak at 0.56V. By the third cycle there was almost no electrochemical activity in the film. These results are indicative of a film with very poor conductivity and switching characteristics.

To improve the cycling of the PTEP, a copolymer was grown from a equimolar mixture of TEP and 3-methylthiophene (3MT). The $E_{p/2}$ for oxidation of 0.01M 3MT in 0.4M TBATFB/ACN was 1.36V (1.71V vs. SCE) at 100 mV/s. The electrodeposition experiment was repeated on the Pt electrode in a solution of 0.1M 3-methylthiophene and 0.1M TEP in 0.4M TBATFB in ACN. Holding the working electrode at 1.5V led to formation of a dark, thick deposit. When thinner films were held at 0.0V for over 5 min, they became light yellow. Cyclic voltammograms of the copolymer films were stable and had comparable charge in the anodic and cathodic portion when cycled at 20 mV/s. $E_{p,a}$ was at 0.62V and $E_{p,c}$ at 0.55V. At 100 mV/s, $E_{p,a}$ and $E_{p,c}$ became 0.75V and 0.50V;

the increase in the $E_{p,a}-E_{p,c}$ value again indicated limiting conductivity in the film. A visible change in the transparency and color of the thin films deposited on these substrates could be detected as it was cycled between 0.0 and 1.5V in 0.1M LiClO₄/ACN without the monomer mixture.

Films of the copolymer were also deposited on 1 cm x 3 cm quartz flats coated with indium tin oxide (ITO). Improved uniformity of the film was obtained if the ITO had a very thin layer of Au deposited on it. We also determined that the film formed more evenly on the ITO from a mixed solvent of dichloromethane (DCM) and ACN in the ratio of 4:1 with tetrabutylammonium hexafluorophosphate (TBAHFP) as the electrolyte [9,10]. Thick, dark films which could be peeled off the substrate were formed by holding the electrode at 2.0V for two minutes. Thinner films which were more suitable for UV-vis analysis were made by 4 cycles between 0.0V and 2.0V at 100 mV/s. A scanning electron micrograph of one of these films is shown in Figure 1. The morphology is globular and relatively porous which is similar to that obtained with 3-octylthiophenes and in contrast to the more compact structure of poly(3-methylthiophene) [11].

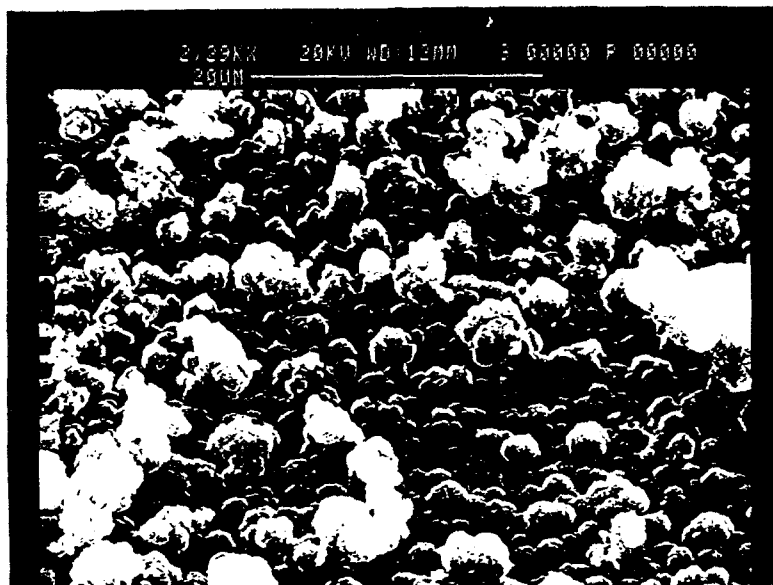


Figure 1. Scanning electron micrograph of 3MTP-TEP copolymer on an ITO substrate formed by four cycles between 0.0V and 2.0V vs Ag/AgNO₃ at 100 mV/s from a solution of 0.4M TBAHFP in 80% DCM/20%ACN.

Photodoping

Measurement of the UV-vis spectra of the electropolymerized films was done on a Hewlett Packard diode array spectrometer, Model 8452A [12]. The ITO substrate was used as the background spectrum. The spectra of the electrochemically reduced and oxidized 3MT-TEP copolymer after holding it at 0.0V and 1.5V vs Ag/AgNO₃, respectively, are shown in Figure 2. As is generally observed with polyalkylthiophenes [13], the broad absorption in the near UV decreases in the

oxidized film as the absorption in the region around 650 increases. The isosbestic point for the copolymer film is about 500 nm. The peaks and valleys separated by about 40 nm evident in the oxidized film are interference fringes from multiple layers of thin films on the glass substrate.

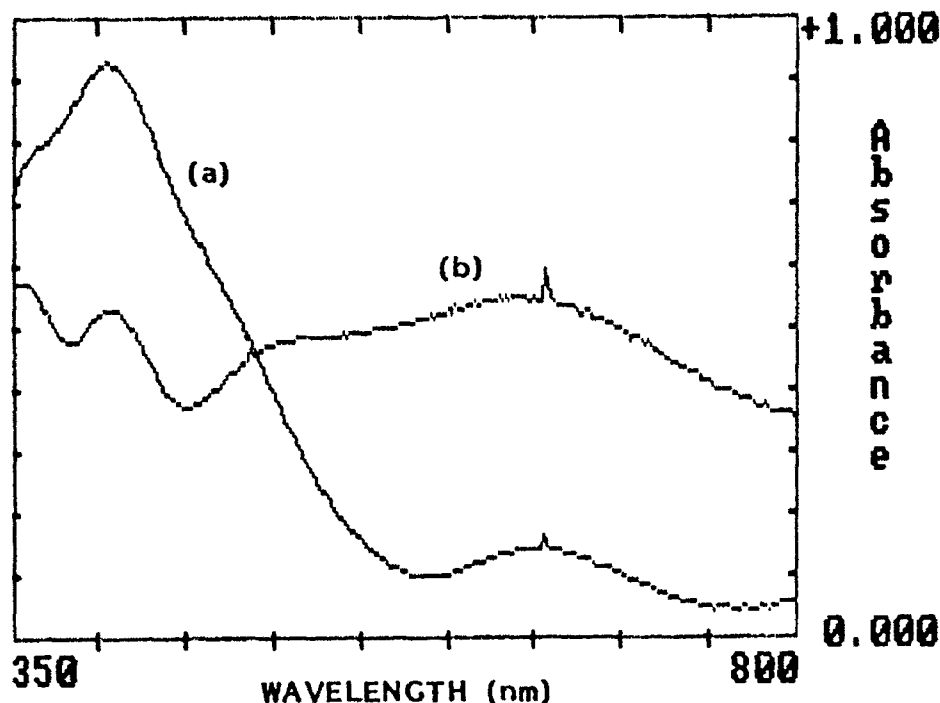


Figure 2. UV-vis spectrum of 3MT-TEP copolymer film on ITO/Au substrate in the reduced (a) and oxidized state. The film was deposited on an ITO/Au substrate by potential cycling. Spectrum of the uncoated substrate was used as the reference.

The spectra in Figure 2 are significantly different from pure poly(3-methylthiophene) which has its maximum in the reduced state at 500 nm and the isosbestic point at 600 nm. The wavelength of the absorbance peak at ≈ 400 nm is closer to that reported for poly(3-heptoxymethylthiophene) ($\lambda_{\text{max}} = 430$ nm) than for poly(3-hexoxyethylthiophene) ($\lambda_{\text{max}} = 525$ nm) [14] and indicates short chain oligomers or strained polymers in agreement with the low conductivity reflected in the electrochemical experiments on the copolymer. The copolymer film in the reduced state was irradiated for 60 min with deep UV (220-260 nm) using an Oriel Illumination System with a 4-1/4" sq uniform beam. The integrated spectral intensity from the 500W Hg/Xe lamp over the wavelength range was 8 mW/cm². After the irradiation, the spectrum was identical to that obtained before the irradiation, demonstrating that the irradiation itself did not cause spectral changes in the film.

To verify that we could observe photodoping of the copolymer, we immersed a reduced copolymer film for 30 min in a saturated solution of diphenyliodonium chloride (DPIC) in methanol. The treatment caused no change in the UV-vis spectrum of the film. The film was then irradiated for 35 minutes. The spectrum before and after the irradiation are given in Figure 3. Comparison of the spectral changes caused by electrochemical oxidation shown in Figure 2 with those in Figure 3 after illumination clearly indicates that some photodoping has occurred. Further illumination and overnight treatment in the DPIC followed by irradiation did not change the spectrum. We surmise that the DPIC did not penetrate the bulk of the film and only the exposed surface layer of the film in contact with the DPIC could be photodoped by this process.

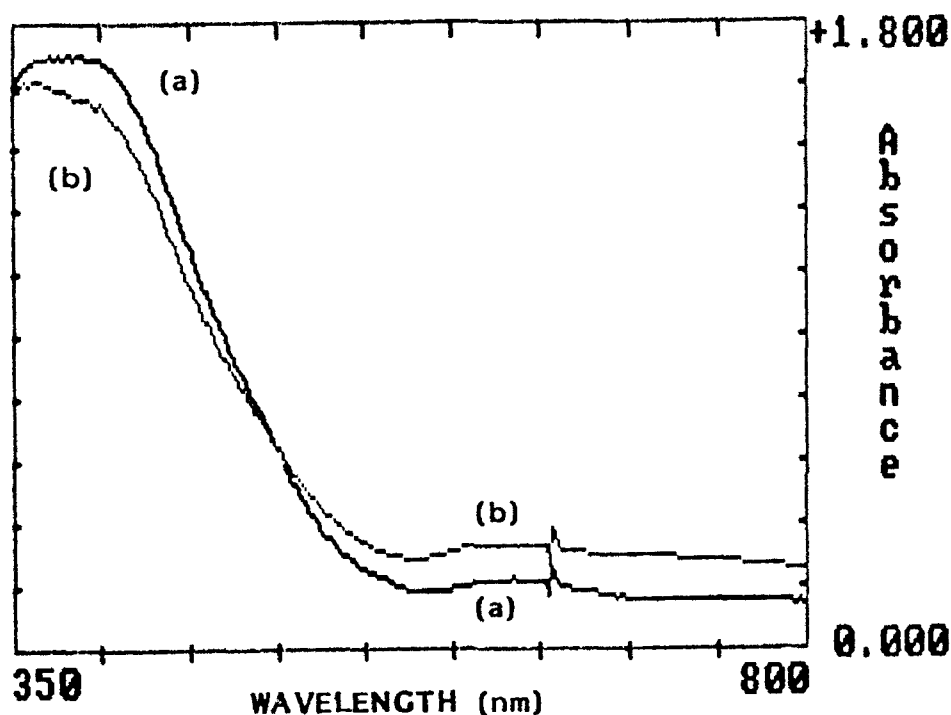


Figure 3. Effect of photodoping by UV illumination of a 3MT-TEP copolymer film that had been immersed in a diphenyliodonium chloride solution: (a) before irradiation, (b) after irradiation.

The reduced copolymer films were immersed in a solution of iodosobenzene diacetate, acetic anhydride and glacial acetic acid in the proportions used by Crivello and Lam [8] to convert anisole to the 4-methoxydiphenyliodonium salt. After immersion for 10 minutes, a drop of sulfuric acid was added. Treatment was also done with the sulfuric acid added before the film was immersed. Exposure of pure PTEP films to this latter solution for periods longer than 5 min visibly degraded the film. With the copolymer films, however, extended treatment could be used without apparent deterioration of the films.

Treatment of the reduced copolymer films with the solution converted the films from a pale yellow to a dark green. The spectral change caused by the treatment is shown in Figure 4. Clearly, the treatment itself led to oxidation of the film. Electrochemical reduction of the treated film partially returned the oxidized film to its reduced state (Figure 4(c)). After a 20 min irradiation of the reduced film, there was no change in the spectrum. From the published spectrum of a range of iodonium compounds [8], if iodonium compound VII had been formed by the treatment, it would have photodissociated and doped the film. Immersing the film overnight in the solution, reducing it electrochemically, and then irradiating it for 60 min also did not produce any indication of photodoping. Based on the experiments with DPIC, we expected to observe some photodoping even if the covalently bound diphenyliodonium moiety had been formed only at the surface of the film. We conclude, therefore, that the reaction with the iodosobenzene diacetate did not produce the desired iodonium compound.

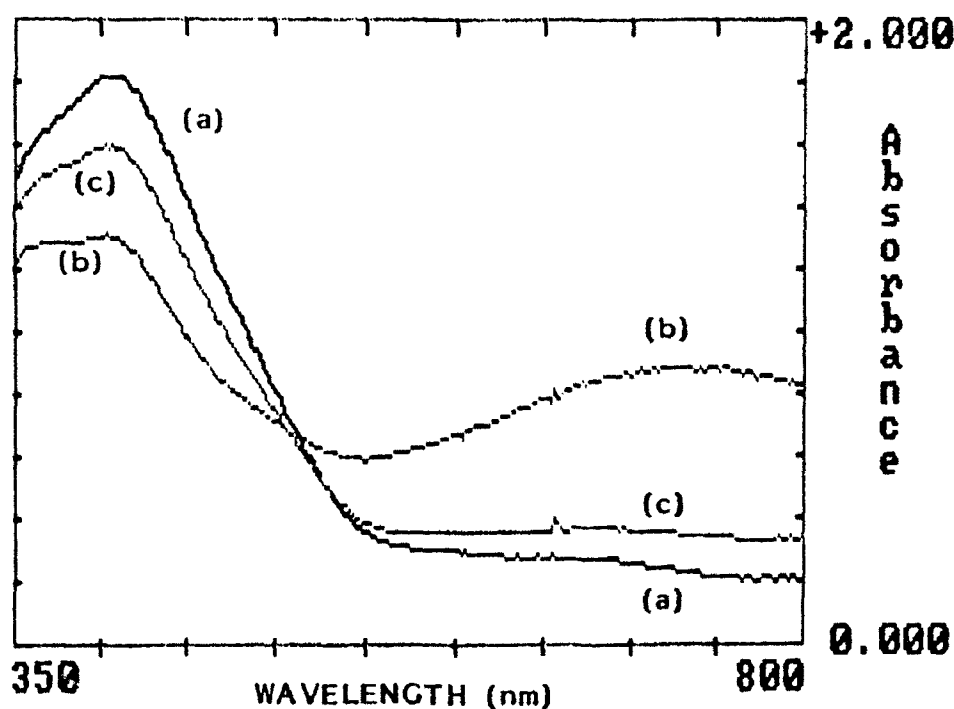


Figure 4. Effect of treatment of the 3MT-TEP copolymer film with the iodosobenzene diacetate solution: (a) before treatment, (b) after 10 min immersion in the solution, (c) after electrochemical reduction.

3-Ferrocenylpyrrole

Some preliminary spectral measurements were made on 3-ferrocenylpyrrole (FcP), VIII, a compound made previously in our laboratory which might show intramolecular photodoping [15].

The electrochemistry of FcP indicated significant interaction between the Fc substituent and the pyrrole. Both ferrocene (Fc) and ferrocenium (Fc^+) have intense absorption bands attributed to ligand \rightarrow metal charge transfer [16] which might induce transient doping in a polymer of FcP.

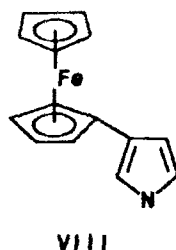


Figure 5 compares the absorption spectrum of FcP with Fc and pyrrole (Py) in methanol. The absorbance maximum of the lowest electronic transition is at 446 nm compared to 442 nm for the unsubstituted ferrocene (Fc) and has an absorption coefficient 2.2 times larger. This small bathochromic shift and enhancement of the oscillator strength agrees with other work on substituted ferrocene derivatives [17]. This electronic transition is primarily associated with the internal d-d transitions in the metal. The absorption at the next shorter wavelength, which has its maximum at 326 nm in Fc, is an order of magnitude stronger in FcP and has a hypsochromic shift of the maximum to 307 nm. This band is associated with a $\pi\text{-}\pi^*$ transition with possible contribution from charge transfer bands. Excitation of this band potentially will be useful in initiating electron transfer reactions for the switching of polymer films into which it is incorporated. Unfortunately, attempts to make polymers of FcP or copolymers with Py by both electrochemical and chemical methods have thus far been unsuccessful [18].

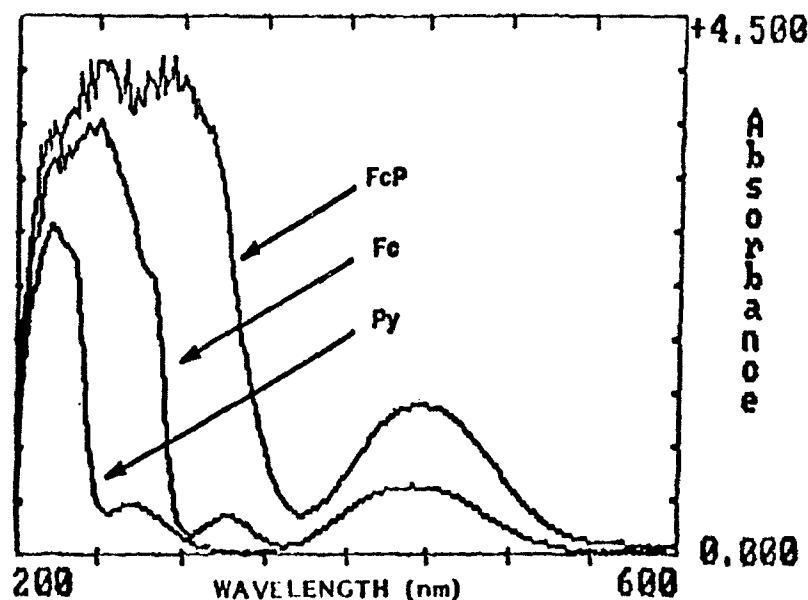


Figure 5. UV-vis absorption spectra of 3-ferrocenylpyrrole (FcP), ferrocene (Fc), and pyrrole (Py) dissolved in methanol.

3.0 CONCLUSION

None of the approaches investigated were successful in synthesizing a polymer with a photoactive substituent that would lead to photodoping of the polymer. Incorporating the chromophore in the monomer appears to be the preferable synthetic route since it eliminates the need for the subsequent treatment of the polymer which itself oxidized the film. Electrochemical reduction of the oxidized film did not return it to the completely reduced state. Attaching the chromophore to the monomer would also ensure the distribution of the chromophore throughout the polymer rather than limiting it to the surface. One anticipated difficulty with polymerizing the substituted monomer is inhibition of the polymerization because of the size of the chromophore. If it is attached through a flexible alkyl chain, the polymerization may be possible. Alternatively, the substituted monomer may be used as one component to form a copolymer. Until these synthetic challenges are overcome, we are unable to determine if the speed, uniformity and stability of the substituted photoswitchable polymer is superior to that obtained with a system made by physically mixing the photodopant in the polymer matrix.

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APPENDIX

PUBLICATIONS:

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